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TITLE: Thermoplastic polyurethane resin mould for e.g. tubes, diaphragm, wet suit or wire cables - consists of a polyurethane film having antimicrobial and antifungal activity coated on the surface of a thermoplastic polyurethane resin mould having predetermined elongation.

PATENT-ASSIGNEE:

ASSIGNEE	CODE
NIPPON MIRACTORAN KK	NIMIN

PRIORITY-DATA: 1997JP-0197780 (July 8, 1997)

PATENT-FAMILY:

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INT-CL (IPC): B32B 27/40; C08J 7/04; C08K 5/16; C08L 75/04

ABSTRACTED-PUB-NO: JP11028797A

BASIC-ABSTRACT:

NOVELTY - An antimicrobial and antifungal polyurethane film having a 100% modulus and tensile strength of 20-200 kgf/cm² is coated on the surface of a thermoplastic polyurethane resin mould. The resin has an elongation of 300-1000%.

DETAILED DESCRIPTION - An INDEPENDENT CLAIM is also included for the production of the antimicrobial, antifungal thermoplastic polyurethane resin mould. A polyurethane film containing the antimicrobial and antifungal agent is applied on the surface of a polyurethane resin mould and then hardened.

USE - The mould is used for tubes, wire cables, diaphragms and wet suits.

ADVANTAGE - The mould has high strength and flexibility, and antimicrobial and antifungal activity.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: THERMOPLASTIC POLYURETHANE RESIN MOULD TUBE DIAPHRAGM WET SUIT WIRE CABLE CONSIST POLYURETHANE FILM ANTIMICROBIAL ANTIFUNGAL ACTIVE COATING SURFACE THERMOPLASTIC POLYURETHANE RESIN MOULD PREDETERMINED ELONGATE

DERWENT-CLASS: A25 A86 A88 D22 P73**CPI-CODES:** A05-G01E; A08-M02; D09-A01;

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PATENT ABSTRACTS OF JAPAN

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(21)Application number : **09-197780** (71)Applicant : **NIPPON MIRACTRAN KK**
(22)Date of filing : **08.07.1997** (72)Inventor : **TAKAHASHI HIROBUMI**
KIKUCHI AKIO
YAMAGISHI AKISHIGE

(54) ANTIBACTERIAL AND MILDEWPROOFING THERMOPLASTIC POLYURETHANE RESIN MOLDING AND ITS MANUFACTURE

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a thermoplastic polyurethane resin molding having a highly stretched polyurethane film including excellent antibacterial and mildewproofing properties, and a method for manufacturing it.

SOLUTION: In the thermoplastic polyurethane resin molding having antibacterial and mildewproofing properties and having an antibacterial and mildewproofing polyurethane film formed on a surface of the resin molding, the polyurethane film contains polyurethane resin having film characteristics of 100% tensile stress of 20 to 200 kgf/cm² and an elongation of 300 to 1000%, antibacterial agent and/or mildewproofing agent. And, the method for manufacturing the resin molding comprises the steps of coating and curing a surface of the thermoplastic polyurethane resin molding with polyurethane paint containing polyurethane resin having the film characteristics of the 100% tensile stress of 20 to 2000 kgf/cm² and elongation of 300 to 1000%, antibacterial agent and/or mildewproofing agent, and forming an antibacterial and mildewproofing polyurethane film thereon.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to antibacterial, the thermoplastic polyurethane-resin moldings which prepared the high extension polyurethane coat of *****, and its manufacture method.

[0002]

[Description of the Prior Art] Conventionally, the method of carrying out melting mixture of antibacterial and the antifungal agent, making the base resin of a TPU moldings contain as common practice most, and fabricating which gives antibacterial and ***** to a thermoplastic polyurethane-resin (henceforth TPU) moldings, and the method of making contain antibacterial and an antifungal agent in a paint, and applying to a TPU moldings are learned.

[0003]

[Problem(s) to be Solved by the Invention] However, by the method of carrying out melting mixture and making the conventional TPU containing antibacterial and an antifungal agent, the heat history at the time of melting mixture was not avoided, but antibacterial and the antifungal agent pyrolyzed, the performance fell, or the property of TPU was spoiled by antibacterial and the antifungal agent, and there were problems -- sufficient performance is not obtained. Moreover, the paint film was lacking in flexibility, there were problems, like a crack tends to enter and the method of making antibacterial and an antifungal agent containing in the conventional paint was inadequate [problems] as a paint for rubber elastic bodies like TPU, when it applied to soft mold goods. this invention solves the trouble of well-known technology such conventionally, and aims at offering antibacterial, the TPU moldings which prepared the high extension polyurethane coat excellent in *****, and its manufacture method.

[0004]

[Means for Solving the Problem] By this invention persons' making specific polyurethane coating contain an antimicrobial agent etc., as a result of inquiring wholeheartedly, and applying, it finds out that antibacterial [outstanding] and the TPU moldings which has ***** and many physical properties are obtained, and came to complete this invention.

[0005] Namely, this invention is the TPU moldings which was excellent in the front face of a TPU moldings at antibacterial, antibacterial [which come to form a ***** polyurethane coat], and ***** , and the aforementioned antibacterial one and a ***** polyurethane coat are the 100% tensile stress 20 - 200 kgf/cm². And it is the TPU moldings which was excellent in the aforementioned antibacterial one characterized by the bird clapper, and ***** from the polyurethane resin which has the film property of 300 - 1000% of elongation, and an antimicrobial agent and/or an antifungal agent

[0006] Moreover, this inventions are antibacterial [above] and the manufacture method of a TPU moldings excellent in ***** , and are the 100% tensile stress 20 - 200 kgf/cm² to the front face of a TPU moldings. And the polyurethane coating containing the polyurethane resin which has the film property of 300 - 1000% of elongation, and an antimicrobial agent and/or an antifungal agent is applied and stiffened, and it is characterized by forming antibacterial and a ***** polyurethane coat.

[0007]

[Embodiments of the Invention] By the thing and the organic system which are generally marketed, the antimicrobial agent used for this invention, and an antifungal agent for example 2-(4-thiazolyl) Benz imidazole (TBZ), 2-(methoxycarbonyl) Benz imidazole, Imidazole systems, such as 2-(Benz imidazole) urethane, 1, 2-Benz iso thiazoline-3-ON, and 2- thiazole systems, such as n-octyl-4-iso thiazoline-3-ON, -- Nitril systems, such as 2, 3, 5, and 6-tetrapod chloro isophthalonitrile, N-(fluoro dichloro methylthio) phthalimide, N, N - Halo alkyl thio systems, such as a dimethyl-N-(dichloro fluoro methyl) thio-N'-phenyl sulfamide, The compound of a pyridine system, a triazine system, a bromine system, a quarternary-ammonium-salt system, and an annular nitrogen system can be mentioned. By the inorganic system, the thing which made heavy metal ions, such as a complex ion, a copper ion, and iron ion, fix to ****, such as calcium phosphate, a zeolite, and a zirconium phosphate, can be mentioned. These antimicrobial agents and an antifungal agent can also be used as two or more sorts of mixture, even if independent.

[0008] the polyurethane resin (henceforth the polyurethane resin for paints) for forming antibacterial and a ***** polyurethane coat in this invention -- 100% tensile stress -- 20 - 200 kgf/cm² it is -- elongation has 300 - 1000% of film property 100% tensile stress is 20 kgf/cm². It is too soft, and is not practical as a coat, and the polyurethane resin for paints of the following is 200 kgf/cm². What is exceeded is too hard, and since it spoils the feeling of TPU, it is not desirable. Moreover, elongation cannot follow a coat in footsteps of elongation of TPU, and less than 300% of polyurethane resin for

paints does not have it. [desirable] The thing exceeding 1000% does not become weak and have a desirable coat. [0009] The polyurethane resin for paints used for this invention is usually manufactured from a polyol, the poly isocyanate, a with a number average molecular weight of 500 or less blended if needed active hydrogen compound, and the solvent and catalyst that dilute these by the case.

[0010] the thing exceeding the number average molecular weight 500 which usually has a hydroxyl group at the end used for the reactional phase hand of the poly isocyanate as a polyol used for manufacture of the polyurethane resin for paints in this invention -- macromolecule polyols, such as a polyether polyol of number average molecular weight 1000-3000, a polyester polyol, a polyester amide polyol, a polyether ester polyol, and a polycarbonate polyol, can be mentioned preferably As a polyether polyol, the graft polymer by polymerization products, such as a tetrahydrofuran, a propylene oxide, and ethylene oxide, or these copolymerization products, and the vinyl monomer of a polyether etc. can be mentioned. As a polyester polyol and a polyester amide polyol, from polyhydric alcohol and multiple-valued carboxylic acids, diamines or amino alcohols are used together by the case, and what is obtained by the condensation reaction is mentioned. As these polyhydric alcohol, ethylene glycol, a diethylene glycol, 1, 4-butanediol, 1, 6-hexandiol, 2-methyl propanediol, neopentyl glycol, 3-methyl-1,5-pentanediol, 2-methyl octanediol, 1, 9-nonane diol, 1, 4-cyclohexane dimethanol, a glycerol, and a trimethylol propane are mentioned, for example. As multiple-valued carboxylic acids, a succinic acid, an adipic acid, a sebacic acid, a dimer acid, a hydrogenation dimer acid, a phthalic acid, phthalic-acid alkyl ester, trimellitic acid, a maleic acid, boletic acid, and an itaconic acid are mentioned, for example. Moreover, what is obtained by the ring opening polymerization of cyclic ester, such as a butyrolactone, a valerolactone, and a caprolactone, is mentioned. What a polyether polyol is used for a part or all of polyhydric alcohol that is used in case a polyether ester polyol manufactures the above-mentioned polyester polyol, and also is obtained like a polyester polyol is mentioned. As a polycarbonate polyol, what is obtained according to the ester exchange reaction of diols, such as 1, 6-hexandiol, 1, and 4-cyclohexane dimethanol, and the annular carbonate like dialkyl carbonate, diaryl carbonate, or ethylene carbonate, for example is mentioned. Moreover, the polyol which introduced the unsaturation machine into a part of these polyols can also be mentioned. These polyols can be used as a prepolymer which is made to react beforehand the poly isocyanate and if needed with a with a number average molecular weight of 500 or less active hydrogen compound, and has a hydroxyl group at the end.

[0011] As a poly isocyanate used for manufacture of the polyurethane resin for paints in this invention Phenylene diisocyanate, tolylene diisocyanate, xylylene diisocyanate, Tetramethyl xylylene diisocyanate, naphthylene diisocyanate, Aromatic diisocyanate, such as diphenylmethane diisocyanate and these isomers, Moreover, 1, 6-hexamethylene di-isocyanate, 1, 12-dodecane diisocyanate, Aliphatic diisocyanate, such as TORIMECHIRU-hexamethylene di-isocyanate, Moreover, cycloaliphatic diisocyanate, such as a cyclohexane diisocyanate, dicyclohexylmethane diisocyanate, isophorone diisocyanate, hydrogenation xylylene diisocyanate, and norbornane-diisocyanate methyl, etc. can be mentioned. Moreover, the poly isocyanate conversion object by the reaction of the isocyanate machine end compounds by the reaction of these compounds and an active hydrogen machine content compound like a trimethylol propane or these compounds, for example, an isocyanuration reaction etc., etc. can be mentioned. Moreover, the poly isocyanate which stabilized the part by the block agent which has active hydrogen, such as a methanol, n-butanol, benzyl alcohol, an ethyl acetoacetate, an epsilon caprolactam, a methyl-ethyl-ketone oxime, a phenol, and cresol, in [one] a molecule can also be mentioned.

[0012] As a with a number average molecular weight of 500 or less active hydrogen compound which can be used for manufacture of the polyurethane resin for paints in this invention Ethylene glycol, a diethylene glycol, a propylene glycol, 1, 4-butanediol, 1, 6-hexandiol, 1, 9-nonane diol, Screw-beta-hydroxy ethoxy benzene, 3-methyl-1,5-pentanediol, Neopentyl glycol, N-phenyl diisopropanolamine, A monoethanolamine, a glycerol, a trimethylol propane, the ethylene oxide of ethylenediamine, Unsaturation machine content glycols, such as an addition product of a propylene oxide and trimethylolpropane methacrylate, and a maleic-acid screw (2-hydroxyethyl ester), etc. can be mentioned.

[0013] A solvent can be used for manufacture of the polyurethane resin for paints in this invention if needed. As such a solvent, ethyl acetate, butyl acetate, a methyl ethyl ketone, a methyl isobutyl ketone, various Cellosolves, toluene, a xylene, a dimethylformamide, dimethyl sulfoxide, and a cyclohexanone are mentioned, for example.

[0014] The catalyst other than a solvent can be further used for manufacture of the polyurethane resin for paints in this invention if needed. As such a catalyst, it is triethylamine, triethylenediamine, N-methyl imidazole, N-ethyl morpholine, 1, and 8-diazabicyclo, for example. - The Lynn system compounds, such as organic metals, such as amines, such as 5, 4, and 0-undecene -7 (DBU), a potassium acetate, stannous octoate, and dibutyltin dilaurate, a tributyl phosphoretted hydrogen, HOSUFOREN, and HOSUFOREN oxide, are mentioned.

[0015] the ratio (R value) of the number [as opposed to / in the polyurethane resin for paints in this invention / according to a polyol, the poly isocyanate, and the need / the total number of active hydrogen machine mols for a with a number average molecular weight of 500 or less active hydrogen compound] of isocyanate machine mols -- desirable -- 0.7-1.5 -- it is blended and manufactured so that it may be set to 0.9-1.3 still more preferably When using together a with a number average molecular weight of 500 or less active hydrogen compound, it blends so that the ratio (R' value) of the number of active hydrogen machine mols of a polyol to the number of active hydrogen machine mols of an active hydrogen compound may become 15 or less preferably.

[0016] The polyurethane coating in this invention can add and prepare an antimicrobial agent and/or an antifungal agent to the aforementioned polyurethane resin for paints. As for an antimicrobial agent and/or an antifungal agent, it is desirable to add 0.1 to 5.0% of the weight to the polyurethane resin for paints (solid content), respectively (independent or all in the case of mixing and blending).

[0017] The polyurethane coating in this invention can carry out 1-20 weight section addition of the isocyanate machine end compound by the isocyanuration reaction of the poly isocyanate etc. to the polyurethane-resin 100 weight section for paints if needed. In this case, catalysts, such as a triethylenediamine and dibutyltin dilaurate, can be added if needed.

[0018] Thixotropy agents [, such as bulking agent; Aerosil], such as an additive further various besides the aforementioned solvent to the polyurethane coating in this invention, for example, a silica, talc, and clay; the leveling agent of silicone and a fluoroiresin system, a defoaming-agent; coloring agent, etc. can also be added and used.

[0019] The polyurethane coating in this invention can be painted as 1 liquid type or a 2 liquid type. As the method of application, a spray method, a brushing method, a immersion coating method, electrostatic spray painting, etc. are employable.

[0020] In this invention, the TPU moldings for forming antibacterial and a ***** polyurethane coat fabricates TPU generally used by common practice, such as an injection-molding method and an extrusion method, and is obtained. A limit of a configuration does not have this TPU moldings at the shape of the shape of the shape for example, of a sheet, and a film, and a belt, and a tube. Before this TPU moldings forms a coat, it is desirable to make the front face of a moldings pure by degreasing etc. Although the front face of this TPU moldings is made to apply and harden polyurethane coating and a coat is made to form in it, you may promote hardening of a coat by heating etc. after an application. As for the thickness of the coat which it is desirable and is made to form, it is desirable that it is 10-1000 microns that the TPU moldings of this invention makes the coat of polyurethane coating form uniformly on the surface of [all] a moldings.

[0021]

[Example] Although an example and the example of comparison explain this invention to a detail further, this invention is not limited at all by these. In an example and the example of comparison, the "section" and "%" mean the "weight section" and "weight %" except for the display of tensile stress and elongation, respectively.

[0022] It is a polyurethane resin for paints to a ball mill with example 1 capacity of 1.5L. NIPPORAN 5196 (: by Japan polyurethane industrial incorporated company -- a film property -- 100% tensile stress 65 kgf/cm² --) APASAIDA AK (product made from SANGI, Inc.) as the 350 sections and an antimicrobial agent for 450% of elongation, and 30% of solid contents The 0.5 sections (it is 0.5% to a resin solid content), As an antifungal agent, the 0.5 sections (it is 0.5% to a resin solid content) and the methyl-ethyl-ketone (MEK) 300 section were taught, SANAIZORU 100 (San-Ai Oil Co., Ltd. make) was processed by the rotational frequency 60 - 90rpm for 48 hours, and 650g of polyurethane coating was obtained. The solid content of this polyurethane coating is 16%, and solution viscosity is 120cp(s) / 25 degrees C. It applied to the TPU (Eby Japan MIRAKU tolan incorporated company590PNAT) sheet (50x50x2mm) and test piece JIS No. 3 dumbbell which fabricated this polyurethane coating beforehand by dipping, it heat-treated for 10 minutes at 80 degrees C after [of air-drying] 30 minutes, and the 15-40-micron coat was formed. About the test piece in which the coat was formed, the antibacterial examination, the ***** examination, and the tension test were performed. The result is shown in Table 1.

[Antibacterial test method] It examined according to antimicrobial-activity test-method [of an antibacterial treatment product] 1 "film contact printing" (the independence specification, the criteria, and the antibacterial treatment product test method of inorganic antimicrobial agent study group establishment, such as silver). That is, the culture medium which diluted the bouillon culture medium with the sterilized pure water 500 times is distributed uniformly, and let this be fungus liquid for inoculation (for the number of micro organisms in 0.5ml to be 1.0 to 5.0x10⁵). The antibacterial treatment test piece (three pieces) and the unprocessed test piece (three pieces) were put into the sterilization petri dish, and 0.5ml of fungus liquid for inoculation was inoculated into the test piece (50x50x2mm) front face, and on it, the covering film (it is cut off and used for the size of 45x45mm from the plastic bag sterilization inspection pack for stomachers) was put, and it covered, and saved under the temperature of 35**1 degree C, and the conditions beyond relative humidity 90% After saving for 24 hours, the bacillus adhering to the film was probed and the number of micro organisms in 1ml of this liquid was measured by agar plate cultivation. Bacilli are Escherichia coli (id dog CHIAKORI IFO 3972) and Staphylococcus aureus (Staphylococcus OLE IFO 12732).

[***** test method] JIS It examined according to Z2911. The error criterion of ***** is as follows.

3: 2 in which growth of a bacillus is not accepted : the [tension test method] JIS that the area of the growth portion of a less than [1/3] 1:hypha exceeds [the area of the growth portion of a hypha] one third It examined based on K7311.

[0023] In example 2 example 1, it carried out like the example 1 except having made the amount of APASAIDA AK and SANAIZORU 100 into 1% to the resin solid content, respectively. The test result is shown in Table 1.

[0024] by the same method as example 3 example 1, as polyurethane coating, the 400 sections, the ethyl-acetate 150 section, the toluene 100 section, the APASAIDA AK3 section (it is 3% to a resin solid content), and SANAIZORU 100 were made into the three sections (it is 3% to a resin solid content), and NIPPORAN N-5236 (: by Japan polyurethane industrial incorporated company -- a film property -- 100% tensile stress 100 kgf/cm [2],%, and 25% of solid contents) was carried out The test result is shown in Table 1.

[0025] In example 4 example 1, an antifungal agent was not used but it carried out like the example 1 except having used ZEOMIKKU AV10D (Product made from Shinagawa Fuel ZEOMIKKU) 0.5% to the resin solid content as an antimicrobial agent. The test result is shown in Table 1.

[0026] In example 5 example 1, an antimicrobial agent was not used but it carried out like the example 1 except having used Preventol A3 (product made from a Beyer) 0.5% to the resin solid content as an antifungal agent. The test result is shown in Table 1.

[0027] The APASAIDA AK1.0 section was carried out as the example of comparison 1E590PNAT100 section, and an

antimicrobial agent, the 1.0 section dryblend of SANAIZORU 100 was carried out as an antifungal agent, it kneaded using the extruder, and the pellet was produced. The sheet (50x50x2mm) and the test piece JIS No. 3 dumbbell were produced with the Copyright (C); 2000 Japan Patent Office \square -N'-phenyl sulfamide, The compound of a \square ro \square al \square , \square examination, and the tens were performed about this test piece. The test result is shown in Table 1.

[0028] With the same method as example of comparison 2 example 1, it is polyurethane coating. the N-5232 (: by Japan polyurethane industrial incorporated company -- film property -- 100% tensile stress 10 kgf/cm², 950% [of elongation], and 40% of solid contents) 350 section, the methyl-ethyl-ketone 300 section, the isopropanol 200 section, the APASAIDA AK1.4 section (it is 1% to a resin solid content), and SANAIZORU 100 were made into the 1.4 sections (it is 1% to a resin solid content), and were carried out. The applied test piece was in the state where a paint film separates from a TPU moldings easily weakly.

[0029]

[Table 1]

	実施例					比較例 1
	1	2	3	4	5	
抗菌試験 (生菌数) 大腸菌 黄色ブドウ球菌	10以下 10	10以下 10	10以下 10	10以下 10	— —	1.2×10^3 2.3×10^4
防かび試験	3	3	3	—	3	2
硬さ (HS)	90	90	90	90	90	90
伸び (%)	540	530	560	550	530	580
引張強さ (kgf/cm ²)	460	440	470	480	470	345

[0030]

[Effect of the Invention] It became possible to offer the TPU moldings which prepared the coat to which it excelled in antibacterial and ***** for the first time by this invention, and intensity and elongation were highly rich in flexibility especially a TPU elastic moldings, and its manufacture method as explained above.

[0031] Antibacterial [of this invention], and the TPU moldings with which the ***** polyurethane coat was prepared, For example, a high pressure hose, a medical-application tube, an oil and a pneumatics tube, the tube for water spray, Tube hose, such as a fire hose, an air mat, a diaphragm, Films, such as a keyboard sheet, synthetic leather, a life jacket, and a wet suit An electric wire and cables, such as power and a telecommunication cable, computer wiring, automobile wiring, and various curl codes In various variant extrusion-molding articles, such as various cordages, various driving belts, and a slip stop, and an injection-molding relation A swivel joint, a dust cover, a pedal stopper, a door-lock striker, Autoparts, such as a bush, a spring housing, a bearing, and vibrationproofing parts, It can be used effective in shoes associated parts, such as a SOL of machine parts, such as various gears, seal packing, a connector, a rubber screen, and a print drum, and sports shoes and the point, and a woman shoes top lift, a roller, an axle-pin rake, a grip, chains, etc.

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CLAIMS

[Claim(s)]

[Claim 1] It is the thermoplastic polyurethane-resin moldings which was excellent in the front face of a thermoplastic polyurethane-resin moldings at antibacterial, antibacterial [which come to form a ***** polyurethane coat], and *****, and the aforementioned antibacterial one and a ***** polyurethane coat are the 100% tensile stress 20 - 200 kgf/cm². And thermoplastic polyurethane-resin moldings which was excellent in the aforementioned antibacterial one characterized by the bird clapper, and ***** from the polyurethane resin which has the film property of 300 - 1000% of elongation, and an antimicrobial agent and/or an antifungal agent.

[Claim 2] It is the manufacture method of a thermoplastic polyurethane-resin moldings excellent in antibacterial [according to claim 1] and *****, and they are the 100% tensile stress 20 - 200 kgf/cm² to the front face of a thermoplastic polyurethane-resin moldings. And the aforementioned manufacture method which is made to apply and harden the polyurethane coating containing the polyurethane resin which has the film property of 300 - 1000% of elongation, and an antimicrobial agent and/or an antifungal agent, and is characterized by forming antibacterial and a ***** polyurethane coat.

[Translation done.]